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HERCULES INC.
LAWRENCE KANSAS. 66044

SUN 143-94

Solventless (Extruded) Powder (N-5) General

COST IMPROVEMENT FOR INHIBITING PROCESS OF MARK 43 GRAIN RECLAMATION OF RUN-OFF ELBA SOLVENT BY DISTILLATION

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SUNFLOWER ARMY AMMUNITION PLANT

TECHNICAL DEPARTMENT INVESTIGATION REPORT

K. D. Cowan SUN 143-94 15 April 1969

Solventless (Extruded) Powder (N-5) General

COST IMPROVEMENT FOR INHIBITING PROCESS OF MARK 43 GRAIN

RECLAMATION OF RUN-OFF ELBA SOLVENT BY DISTILLATION

DIGEST

OBJECTIVE

The objective of this study was to provide laboratory data for determining the feasibility of reclaiming run-off ELBA by distillation.

SUMMARY AND CONCLUSIONS

Distillation of run-off ELBA is a practical and economical method of solvent recovery. Results of this study show that distillation of run-off ELBA can be performed safely.

Run-off ELBA, which contained approximately 0.1 percent nitrates calculated as nitroglycerin, 0.8 percent water, 0.9 percent non-volatiles, and 59 percent butyl acetate, was reclaimed by vacuum and ambient pressure distillations. Analyses of distillates from both methods showed no NG as a distillation product. Residue analyses from both types of distillation showed that nitrate content in the residue increased as the residue volume decreased. However, vapor phase analyses showed decomposition and gaseous evolution of

nitrate compounds during distillation. The decomposition rate was temperature dependent and not considered hazardous where the pot temperature did not exceed 161°C.

Approximately 99 percent of the water in a 5 percent water in virgin ELBA mixture was recovered in the first 21 percent of the total volume distilled by ambient pressure distillation. This first portion distilled as an azeotropic mixture of water and butyl acetate (BA), and exhibited an immediate phase separation. Approximately 97 percent of the total water content was in the water phase of this separation and 2.5 percent was in the BA phase.

Cracking studies were conducted on samples of run-off ELBA containing 0.1 percent NG and virgin ELBA containing 0.3 percent NG, to monitor decomposition of the nitrate ester. Vapor analyses indicated that decomposition was occurring. This observation was supported by studies reported in the literature, and by the behavior of the residue which turned yellow after one hour of refluxing and progressively turned darker. Residue analyses did not show a significant amount of decomposition but the test method used was not considered sensitive enough to detect small changes in nitrate content.

Nitrate analyses of separate samples of ELBA containing only NC, 2-NDPA, and EC tape showed that NC, as well as NG, significantly contributes to the total nitrate content in run-off ELBA.

Heat stability tests were conducted on residues from both types of distillations after approximately 90 percent recovery. A vacuum distillation residue containing 2.2 percent NG showed a positive evolution of oxides of nitrogen as determined by a color change of methyl violet paper after 92 minutes at 120°C and after 61 minutes at 134.5°C. (Standard N-5 propellant produces a color change after 45 minutes at 120°C and 19 minutes at 134.5°C.) An ambient pressure distillation residue tested at 161°C produced no color change in methyl violet paper after 1 hour.

Previous work showed that ignition by impact, friction, and explosive propagation of ELBA containing less than 75 percent NG requires energy levels greater than the detecting limits of standard test equipment.

RECOMMENDATIONS

As a result of laboratory success in reclaiming run-off ELBA by distillation, it is recommended that the test objective be expanded to include a production operation. Ambient pressure distillation is recommended in preference to vacuum distillation because of more efficient distilling time and lower equipment costs. The following conditions are recommended for the operation.

1. Batch Operation - Table I shows the approximate material balance expected for an initial charge of 100 gallons of run-off ELBA.
2. Distillation Fractions
    a) First fraction is to be taken at 5 to 10 percent of original

charge volume. Allow the fraction to separate into a water phase and BA phase.

1. Water phase - to waste.

2. BA phase - recycle with subsequent charges.

b) The second fraction containing 80 percent of the original charge volume is to be analyzed for BA by specific gravity and sufficient BA or EL added so that distilled material conforms to virgin ELBA requirements. This material is to be sent to storage for reuse.

c) Final Residue. Approximately 5-10 percent of the original charge volume will be left as residue to be destroyed at the burning grounds.

3. The distillation pot temperature should not exceed 161°C.

INTRODUCTION

In the inhibiting process of the Mark 43 grain, ELBA solvent (a mixture of 35 percent ethyl lactate and 65 percent butyl acetate by volume) is used to bond the ethyl cellulose inhibiting tape to the propellant grain. The control of this process is critical in the production of acceptable grains.

Reuse of run-off ELBA is limited without first upgrading the solvent to approach the purity of virgin ELBA. Several corrective methods considered, including air sparging[1], a process which was successful in removing water from run-off ELBA but not other contaminants, have proven only partially successful.

The volume of ELBA used at Sunflower, the cost of the solvent, and a single source for ethyl lactate make maximum utilization of run-off ELBA desirable. Based on a review of the economics of ELBA from January 1966 to November 1968 and a forecast of expected grain production for 1969, assuming no change in production methods, run-off ELBA is estimated to have a production cost of $150 to 180 thousand per year[2].

To achieve maximum utilization, the development of a practical, efficient, solvent recovery system is required. This report summarizes the results of a laboratory scale investigation for reclaiming run-off ELBA by vacuum and ambient pressure distillation. Recovery yields, distillate and residue composition, residue stability and sensitivity, and water-ELBA separation are discussed.

TERMS AND ABBREVIATIONS

ELBA	A mixture by volume of 35 percent ethyl lactate and 65 percent butyl acetate.
NG	Nitroglycerin
NC	Nitrocellulose
2-NDPA	2-Nitrodiphenylamine
EC	Ethyl Cellulose
$TiCl_3$	Titanium trichloride, a reagent used in the titrimetric method of analyzing nitrate content of nitrate esters. This method is used at Sunflower to determine the nitroglycerin content in propellant.
KF	Karl Fischer method of determining moisture content in organic compounds.
Run-off ELBA	Used in conjunction with ELBA which is not retained by the propellant grain when applied in the inhibiting process.
IR	Infrared
Residue	Refers to any ELBA and non-volatiles left in the distilling flask.
EL	Ethyl Lactate
BA	Butyl Acetate
Modified Heat Stability	Stability tests performed at temperatures other than 120°C and 134.5°C.
KCl	Potassium Chloride
Nitrates	Oxides of nitrogen calculated as NG in all analyses.
Non-Volatiles	All residue material remaining after evaporation to dryness at 100°C.

EXPERIMENTAL

This section describes the apparatus and the procedures used in this investigation. The equipment and the methods of analysis used were those available at the Sunflower Laboratories.

Vacuum Distillation

The vacuum distillation study was conducted first because it was considered less hazardous to perform the distillation at lower temperatures than those required for an ambient pressure distillation.

The vacuum distillations were conducted with the apparatus illustrated in Figure 1. The system consisted of a 3000 ml round bottom flask which was heated by a hot plate in the first trial, but in subsequent trials was heated by a steam bath. Other components in the system were two 1000 ml receiving flasks immersed in an ethylene glycol-water cold bath (12-30°F), a manometer, and a Welch Duo Seal vacuum pump. The distillations were performed at 1 mmHg pressure and a pot temperature range of 80°C to 100°C.

Run-off ELBA was analyzed for nitrates, water, and butyl acetate content. The distillate was analyzed for nitrates, water, and butyl acetate. The final residue was analyzed for nitrate content. A dibutyl phthalate trap, placed in the system to absorb evolved oxides of nitrogen, was sampled after completion of the distillation and analyzed for nitrate content. Qualitative IR spectra were made of the final distillate and residue and compared to reference spectra. Stability tests were conducted on the final residue (see Stability Studies).

Ambient Pressure Distillation

The ambient pressure distillations were conducted in the apparatus illustrated in Figure 2. This apparatus consisted of a distilling flask, an Allihn condenser, a receiving flask, and a 0.3 percent KCl vapor trap attached to an aspirator by which a positive vapor flow was obtained.

An original sample of run-off ELBA was analyzed for nitrates, water, non-volatiles, and BA content and then distilled to 40 percent volume recovery. The distillate and residue were sampled at intervals of 10 percent of remaining volume. The distillates were analyzed for nitrates, water, and BA content and the residues for nitrates and non-volatiles.

Two additional distillations were carried to 90 percent recovery without interruption. The final distillates were analyzed for nitrates and BA content, and the final residues were analyzed for nitrates. Qualitative IR spectra were obtained for these final distillates and residues, and compared to reference spectra. Stability tests conducted on the final residues are described in the section on stability studies.

Related Studies

1. Water-ELBA Separation by Distillation

A mixture of 5 percent water in virgin ELBA was distilled at ambient pressure. Distillate samples taken at 21, 67, 85, and

96 percent recovery were analyzed for water and butyl acetate content.

2. Thermal Cracking

A 2000 ml sample of used ELBA and a 2000 ml sample of 0.3 percent NG in virgin ELBA were each refluxed in the apparatus illustrated in Figure 3. The apparatus consisted of a 3000 ml 3 necked flask, an Allihn condensor, and a 0.3 percent KCl vapor trap connected to an aspirator which supplied a positive vapor flow. Samples were taken from the residue and the KCl trap after 0.5, 1, 1.5, 2.5, 3.5, 4.5 hours refluxing. The residues and trap samples were analyzed for nitrates. Potassium Iodide-starch (KI) papers were periodically placed in the vapor stream to detect the presence of gaseous oxides of nitrogen.

3. Total Nitrate Analysis

Three samples of virgin ELBA with dissolved NC, 2-NDPA, and EC tape respectively, were analyzed for nitrate content by $TiCl_3$ titration to determine if these constituents made any contribution to the total nitrate content in run-off ELBA.

4. Stability Studies

Modified heat stability[3] tests were performed on residue samples from both type distillation studies. Vacuum distillation residues were subjected to temperatures of 120°C and 134.5°C, and the time required for methyl violet paper to indicate a positive test for nitrates was observed. Other vacuum distillation residues were heated to 161°C with observations made at 140°C, 151°C, and 161°C. An IR spectra was made of one vacuum distillation residue which had been subjected to temperature of 161°C for 4 hours.

DISCUSSION OF RESULTS

The results of the vacuum distillation, ambient pressure distillation, related studies and stability tests are discussed in this section.

Vacuum Distillation

Analyses of distillate fractions (Table II) revealed no nitrates. Residue analyses showed that the nitrate concentration was proportional to the volume distilled. There was evidence of decomposition of NG during the distillation as traces of nitrates were detected in the dibutyl phthalate trap and the vacuum pump oil. This observation of decomposition agrees with Naoúm[5], who stated that pure NG cannot be distilled without decomposing, even under vacuum.

There was no distinct water separation during the distillation, however, the initial distillate fraction contained most of the water (see Water-ELBA Separation by Distillation Discussion). Figure 4 shows the BA-EL content of the distillate as a function of volume distilled.

Infrared spectra of typical run-off ELBA and of the final distillate (Figures 5 and 6) showed no differences when compared to spectra of virgin ELBA. The IR spectra of the final residue (Figure 7) indicated that it was composed largely of EL, with EC and nitrate compounds also present.

Modified heat stability tests performed on the residue indicated

the ambient pressure distillations could be safely performed (see Stability Studies).

Ambient Pressure Distillation

The ambient pressure distillation results were similar to the vacuum distillation results. The distillates showed no nitrates (Table III) and the concentration of nitrates and non-volatiles in the residue was proportional to the percent of the total volume distilled. Decomposition of the nitrate esters was evidenced by an immediate change of KI paper when placed in the vapor stream, and by traces of nitrates detected in the KCl trap.

Infrared spectra of the final distillates and residues (Figure 8) were similar to those of the vacuum distillation. These spectra showed that no nitrates were in the distillates, but they were present in the residues.

A 90 percent ambient pressure distillation recovery was accomplished at a maximum pot temperature of 160°C and a vapor temperature of 153°C with a pronounced water separation in the first 10 percent volume fraction. Figure 9 shows the vapor phase composition as a function of temperature.

Related Studies

1. Water-ELBA separation Distillation

An ambient pressure distillation was performed on a 5 percent water in virgin ELBA mixture to determine ease of separation of water and ELBA. Approximately 99 percent of the water was recovered in the first 21 percent of the mixture distilled. Subsequent fractions each contained less than 0.5 percent water.

The water was recovered as an azeotropic mixture which underwent phase separation upon standing. Water which separated from the BA phase made up almost 97 percent (Table IV) of the total water in the original mixture. The BA phase contained 2.45 percent water with the remaining water in the residue. Figure 10 shows the BA content of the ELBA distillate as determined by specific gravity.

2. Thermal Cracking

Samples of run-off ELBA containing 0.1 percent NG, and virgin ELBA containing 0.3 percent NG were each refluxed for 4.5 hours to try to crack the nitrate ester bond. Decomposition of nitrate compounds in the ELBA was verified by detection of nitrates in the KCl trap and by an immediate reaction of KI paper when placed in the vapor stream. A gradual reduction of the nitrate content (Table V) in the residue was not demonstrated due to lack of sufficiently accurate test methods for measuring small changes in concentrations of nitrates.

After approximately 1 hour of refluxing at a pot temperature of 127°C, the ELBA developed a yellow color which became progressively darker until termination of heating at 4.5 hours. This coloration was not observed in the distilling of pure ELBA and it was assumed to be the result of NG decomposition. Naoúm[5] reported a red color developed in pure NG at 135°C and listed the cause as being products of thermal decomposition.

3. Total Nitrate Analysis

Samples containing 0.9 grams of NC, 2-NDPA, and EC tape were dis-

solved in separate 100 ml volumes of virgin ELBA, and each was analyzed for nitrate content. The nitrate content calculated as NG was found to be 0.3 percent for NC, 0.0 percent for EC, and 0.02 percent for 2-NDPA. This study provides evidence that NC contributes significantly to the nitrate content (calculated as NG) in run-off ELBA.

4. Stability Studies

Modified heat stability tests were conducted on residues from both types of distillation to study thermal stability. Residue samples containing 3 ml each from the vacuum distillation were tested at 120 and 134.5°C and the times to a color change of methyl violet paper (Table VI) were observed. The sample tested at 120°C produced only a partial color change after 92 minutes. The sample tested at 134.5°C produced only a partial color change in methyl violet paper after 61 minutes. (Standard N-5 propellant produces a complete color change in the paper after 45 minutes at 120°C and after 19 minutes at 134.5°C). One residue specimen left at 134.5°C for 22 hours evaporated to one half its original volume and changed color from red-brown to dark brown, indicating decomposition.

A vacuum distillation residue sample which analyzed as 2.2 percent NG, was heated from room temperature to 161°C in an oil bath. Observations made at 140, 151, 161°C were as follows: At 140°C no paper change occurred but the sample exhibited a mild boiling action which subsided after about 5 minutes. At 151°C boiling

resumed and a partial color change was seen, but after 20 minutes the color change was still incomplete. At 161°C no further change occurred for 1 hour. At this time a fresh methyl violet paper was inserted and the test was continued. The fresh paper was approximately one half changed after 30 minutes. After 4 hours at 161°C the sample was qualitatively analyzed by IR (Figure 11) and showed no nitrate peaks.

Run-off ELBA has been evaporated to dryness at 100°C by steam bath and oven in routine laboratory determinations of percent total non-volatiles. This analysis has been performed for 3 years with no incident of residue burning.

5. Sensitivity Tests

Previous work[6] reported that ignition by impact and friction, and explosive propagation of ELBA containing less than 75 percent NG (Table VII) requires energy levels greater than the detecting limits of standard test equipment.

6. Resistance to Ignition

It was reported in a separate investigation of NG migration in EC tape, that EC tape containing 14 percent NG could not be ignited in an ambient pressure air atmosphere in a bomb calorimeter.[7] NG in EC tape is essentially the composition of the non-volatiles in the distillation residue.

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3. MIL-STD-286B, Method 404.1.2, "Heat Stability," December .. 1967.

4. MIL-STD-268A, Method 208.1.3, "Nitrates Esters," August 18, 1961.

5. Nitroglycerin and Nitroglycerin Explosives, P. Naoúm, Williams Wilkins Co., 1923, Page 108-136.

6. Holden, G. F., "Sensitivity Evaluation of NG Contaminated 35/65 ELBA (Ethyl Lactate/Butyl Acetate) Mixture," Hercules Incorporated ABL, January 10, 1967, and personal communications with L. S. Treadwell, Hercules Incorporated/ ABL, April 11, 1969.

7. Cathey, J. R., Unpublished Report, "NG Migration in Ethyl Cellulose Tape," Hercules Incorporated/SAAP. Work performed in 1968.

8. Richardson, R. H., "Sensitivity Aspects of Nitroglycerin Casting Solvents," Hercules Incorporated/ABL, April 1, 1964.

TABLE I

APPROXIMATE MATERIAL BALANCE FOR AMBIENT PRESSURE BATCH DISTILLATION

Maximum Pot Temperature of 160°C (320°F)

MATERIAL	INITIAL CHARGE (gal)	DISTILLATE (gal)		RESIDUE (gal)	PROCESS LOSS EVAPORATION (gal)
		WATER PHASE (a)	ELBA (b)		
			(90 percent recovery)		
BA	59		56.5		2.5
EL	39.3		33.3	6.0	
Water	0.8	0.6 (d)	0.2		
Non-Vol(c)	0.9(c)			0.9(c)	
Total	100.00	0.6	90(e)	6.9	2.5

NOTES:

(a) To waste

(b) To storage

(c) Non-volatiles; 0.09 percent by weight made up of EC tape, 2-NDPA, NC, NG.

(d) Represents water distilled in first 5 to 10 percent fraction. Approximately 0.55 gal. will separate as a water phase which will go to waste. Approximately 0.05 gal. will remain in the BA phase which will be recycled with subsequent charges.

(e) The recovery rate (efficiency) diminishes beyond 90 percent volume recovery.

TABLE II

COMPOSITION OF DISTILLATE AND RESIDUE FOR

VACUUM DISTILLATION OF RUN-OFF ELBA

PERCENT TOTAL VOLUME DISTILLED	PRESSURE mmHg	POT TEMP. °C.	VAPOR TEMP. °C.	RESIDUE	DBP TRAP	DISTILLATE		
				NG (b) PERCENT	NG (c) PERCENT	NG PERCENT	WATER PERCENT	BA (a) PERCENT
0				0.1	0.0			
25	1	83	26		0.0	0.0	1.72	82.2
36	1	82	25		0.0	0.0		
60	1		33		0.0	0.0	0.46	73.3
87	1	100	32		0.0	0.0		
94	1	98	50	2.2	0.0	0.0		63.0
Original sample: NG 0.1 percent, Water 0.8 percent, Non-Vol 0.9 percent, BA 59 percent								

NOTES:

(a) Values represent an average of 3 distillations which were performed under the same conditions.

(b) The method used for NG determination was $TiCl_3$ titration. This method is not considered accurate enough for the small quantities involved. The presence of nitrates were shown and trends indicated but values were not considered quantitative.

(c) Nitrates were indicated by the $TiCl_3$ method in the DBP trap, but values obtained were less than the accuracy of the test.

TABLE III

COMPOSITION OF RESIDUE AND DISTILLATE FOR AMBIENT

PRESSURE DISTILLATION OF RUN-OFF ELBA

PERCENT TOTAL VOLUME DISTILLED	POT TEMP. °C.	VAPOR TEMP. °C.	RESIDUE[a]		DISTILLATE[a]		
			NG (b) PERCENT	NON-VOL PERCENT	NG PERCENT	WATER PERCENT	BA PERCENT
0	23	23	0.1	0.92			
10	125	100	0.1	0.97	0.0	2.2	94.6
19	128	121	0.1	1.33	0.0	0.2	85.5
28	132	125	0.1	1.03	0.0	0.2	87.5
34	138	128	0.1	2.36	0.0	0.2	88.1
40	140	128	0.1	1.29	0.0		
90	160	153	1.0		0.0		63
KCl TRAP NG CONTENT .007 ppm							
Original sample: NG 0.1 percent, Water 0.8 percent, Total Vol 0.9 percent, BA 59 percent							

NOTES:

(a) Table represents an average of 2 distillations performed under the same conditions. The first distillation was performed in steps, monitoring the distillate and residue at the fractions listed. The second distillation was conducted without interruption to 90 percent recovery.

(b) This method used for NG determinations was a $TiCl_3$ titration. This method is not considered accurate enough for small quantities involved. The presence of nitrate can be shown and trends indicated, but are not considered quantitative.

TABLE IV

DISTILLATION OF A SYNTHETIC MIXTURE OF
5 PERCENT WATER IN VIRGIN ELBA

PERCENT TOTAL VOLUME DISTILLED	TEMPERATURE RANGE AT WHICH SAMPLE WAS DISTILLED		DISTILLATE			
	POT TEMP. °C	VAPOR TEMP. °C	WATER PERCENT	Sp Gr	BA PERCENT	EL PERCENT
ORIGINAL SAMPLE			5.1[b]	0.9254	63.2	31.7
21.2[a]	98-130	89-109	2.5/96.5	0.8851	92.1	5.5
67.8	133-142	118-121	0.43	0.9092	76.7	22.9
85.8	141-151	124-132	0.38	0.9456	53.2	46.4
96.0	151-161	132-142	0.08			

NOTES:

(a) The first 21 percent distilled separated into 2 phases. The lower (97 percent water) phase accounted for approximately 5 percent and the upper (BA plus 2.5 percent water) phase for about 16 percent of the total volume.

(b) The sample contained 5.1 percent water and 94.9 percent virgin ELBA.

TABLE V

DECOMPOSITION OF NG IN ELBA BY THERMAL CRACKING

TIME HOURS	POT TEMP. °C.	VAPOR TEMP. °C.	NG CONTENT			
			RUN-OFF ELBA		SYNTHETIC MIXTURE (ELBA-NG)	
			RESIDUE[a] PERCENT	KCl TRAP[b] PPM	RESIDUE PERCENT	KCl TRAP PPM
0	23	23	0.1		0.3	
0.5	100-102	40-42	0.1	0.00	0.1	0.00
1.0	125	87	0.1	0.00		
1.5	126	88	0.1	0.06	0.0	0.06
2.5	126	88	0.1	0.12	0.1	0.17
3.5	126	88	0.1	0.11	0.2	
4.5	127	89	0.1		0.3	0.49

NOTES:

(a) NG content was determined by $TiCl_3$ titration.

(b) NG content in KCl traps was determined by a spectrophotometric method using diphenyl benzidine in sulfuric acid as the indicator.

TABLE VI

STABILITY STUDIES OF DISTILLATION RESIDUES BY MODIFIED HEAT STABILITY TESTS

TEMPERATURE °C	TIME TO COLOR CHANGE IN METHYL VIOLET PAPER, MINUTES	
	VACUUM DISTILLATION RESIDUE	AMBIENT PR. DISTILLATION RESIDUE
120	92 PARTIAL CHANGE	
134.5	61 PARTIAL CHANGE	
134.5	54(c)	
140 (a)	RESIDUE BOILED NO CHANGE	
151 (a)	20 SLOW PARTIAL CHANGE	
161 (a)	30 SLOW PARTIAL CHANGE	NO CHANGE AFTER 60 MIN(b)
	STANDARD N-5 PROPELLANT	
120	45 MINUTES	
134.5	19 MINUTES	

NOTES:

(a) Residue samples were heated in an oil bath. Changes were observed in the methyl violet paper as the temperature increased. At 140°C boiling occurred but no paper change was seen. At 151°C boiling reoccurred, and there was a slow partial change in paper color that took place for about 20 minutes. A new methyl violet paper was inserted after the temperature had been at 161°C for about 1 hour. A slow partial change which took about 30 minutes was observed.

(b) Both methyl violet and KI paper were used.

(c) A 4 ml sample was used instead of a 3 ml sample.

TABLE VII

SENSITIVITY VALUES FOR NG-ELBA SOLUTIONS

TEST	UNITS	NG CONTENT PERCENT	INITIATION LEVEL	REF.
IMPACT	ft. lb/in^2	100 70	5.5 63	6,8 6
FRICTION	psi @8 ft/sec	100 78	10 34,800	8 6
THIN FILM PROPAGATION (2mm)	meters per second	100 75	876 No propagation	8 6
Flash Point	°C	0	29	6

VACUUM DISTILLATION APPARATUS

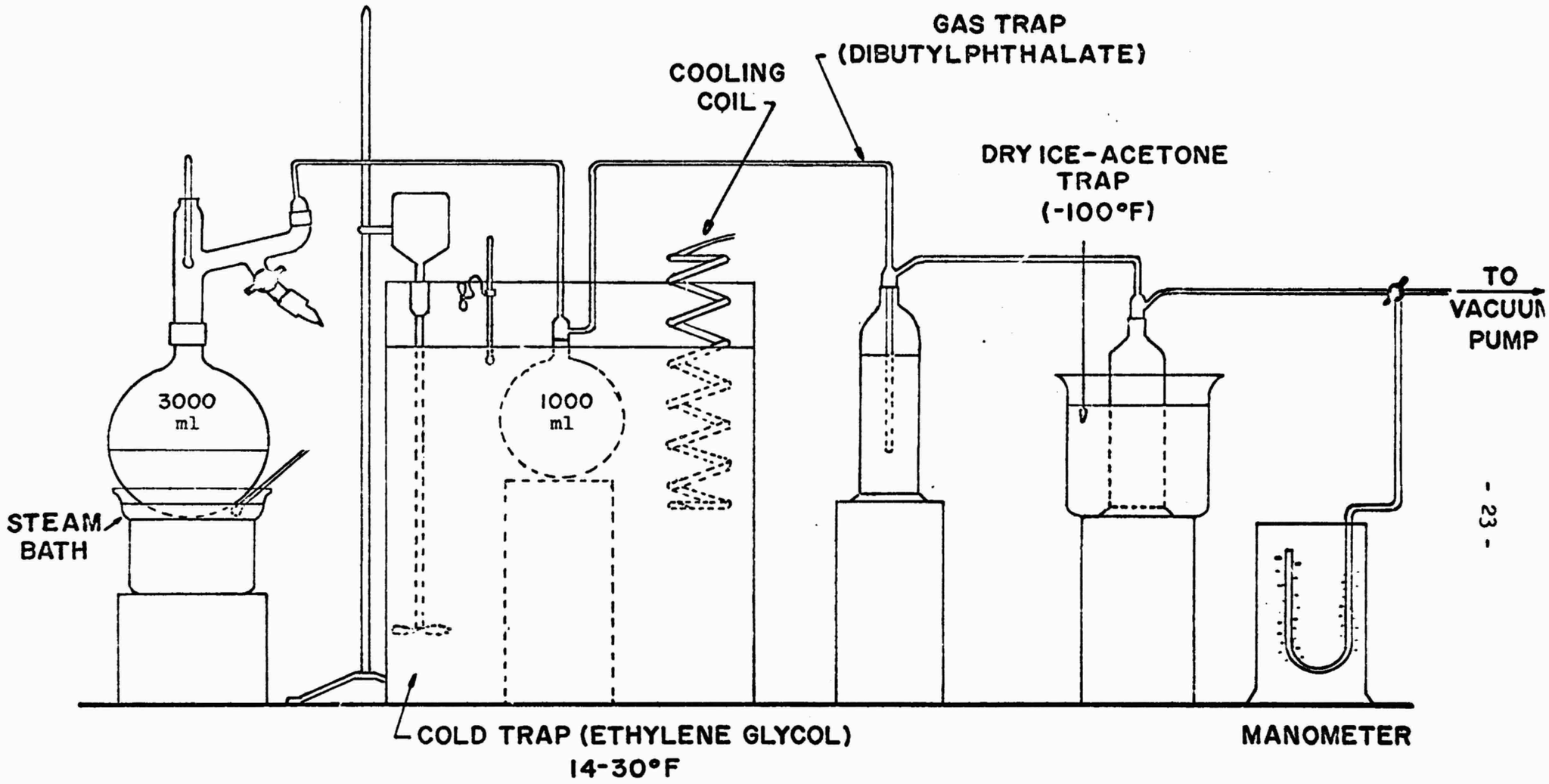


FIGURE I
APPARATUS FOR VACUUM DISTILLATION OF RUN-OFF ELBA

AMBIENT PRESSURE DISTILLATION APPARATUS

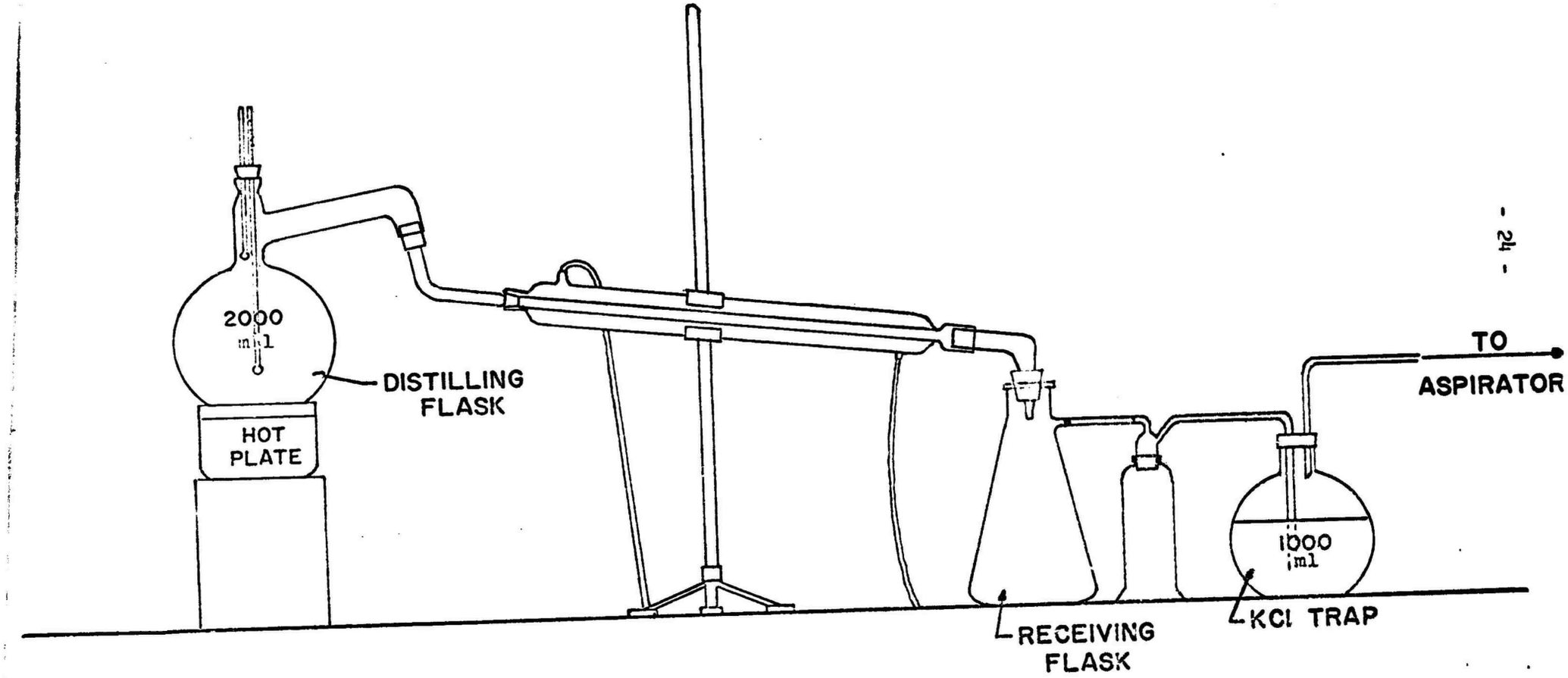


FIGURE 2
APPARATUS FOR AMBIENT PRESSURE DISTILLATION OF RUN-OFF ELEA

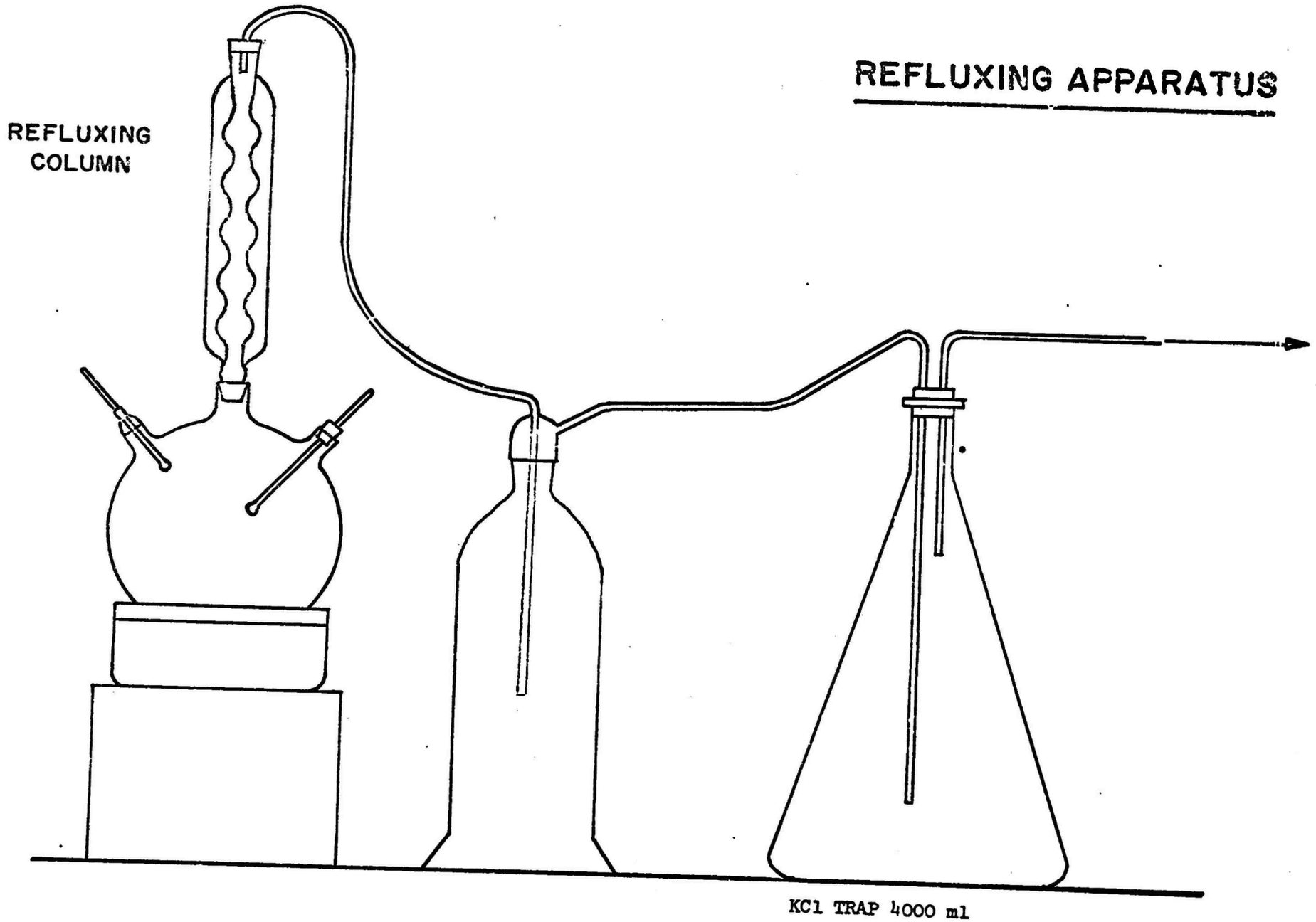


FIGURE 3
APPARATUS FOR THERMAL CRACKING OF NG IN ELBA

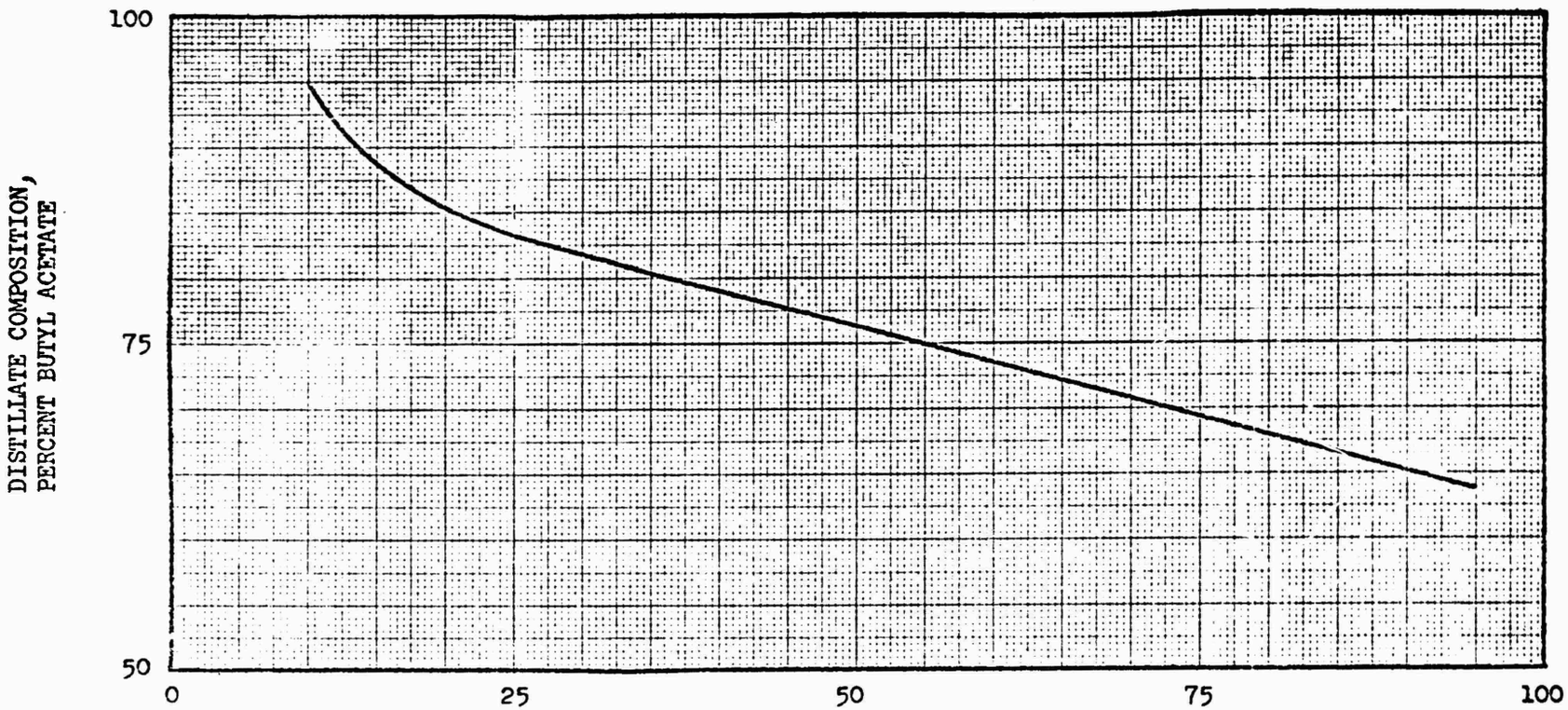


FIGURE 4

BUTYL ACETATE CONTENT IN DISTILLATE AS A FUNCTION
OF THE AMOUNT OF ORIGINAL VOLUME DISTILLED

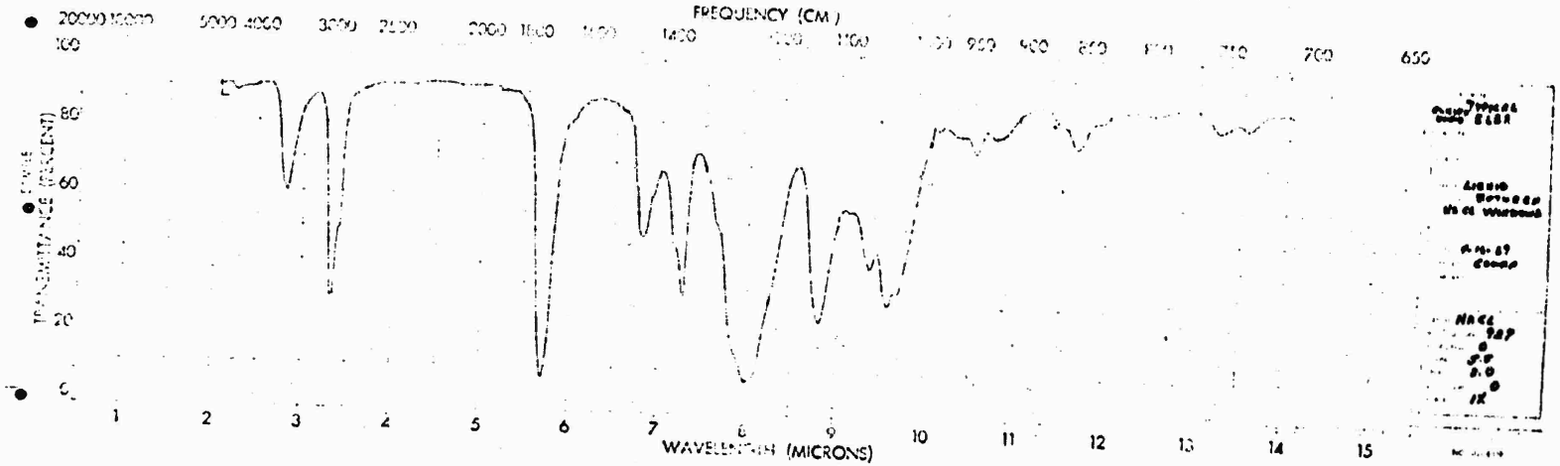
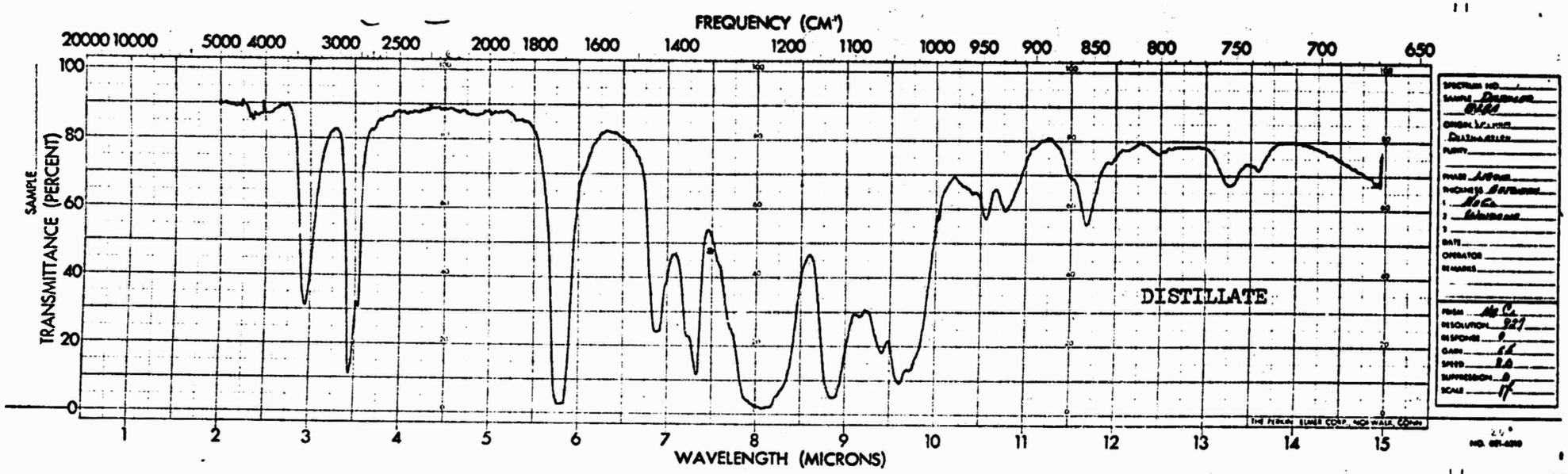


FIGURE 5

QUALITATIVE IR SPECTRA FOR TYPICAL RUN-OFF ELBA



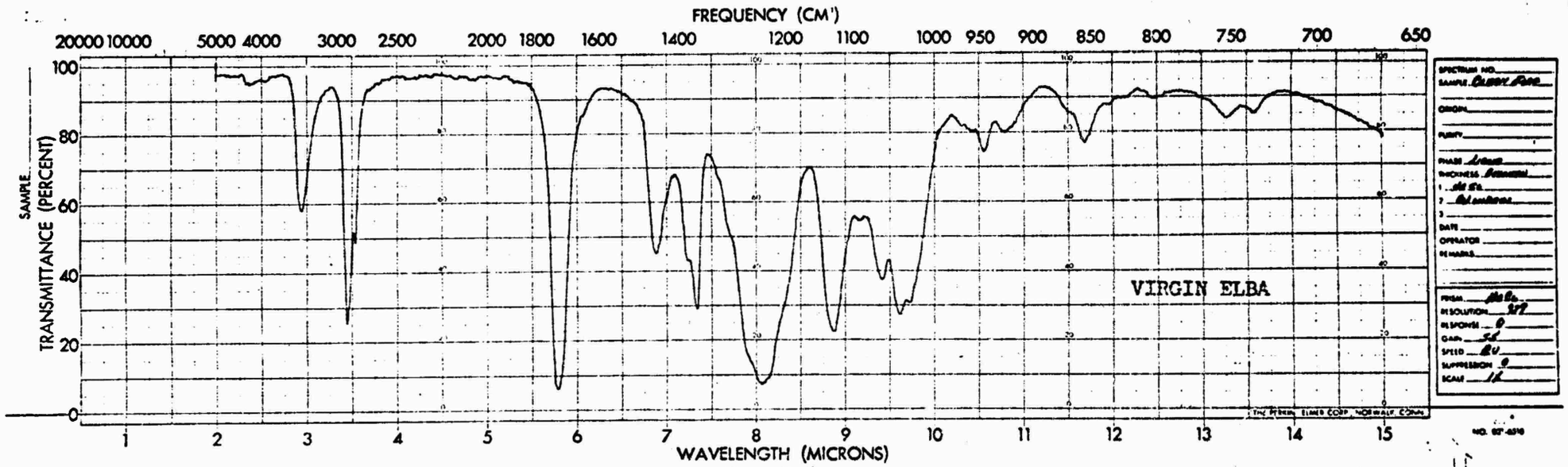


FIGURE 6

QUALITATIVE IR SPECTRA FOR VACUUM DISTILLATION DISTILLATE COMPARED TO REFERENCE SPECTRA OF VIRGIN ELBA

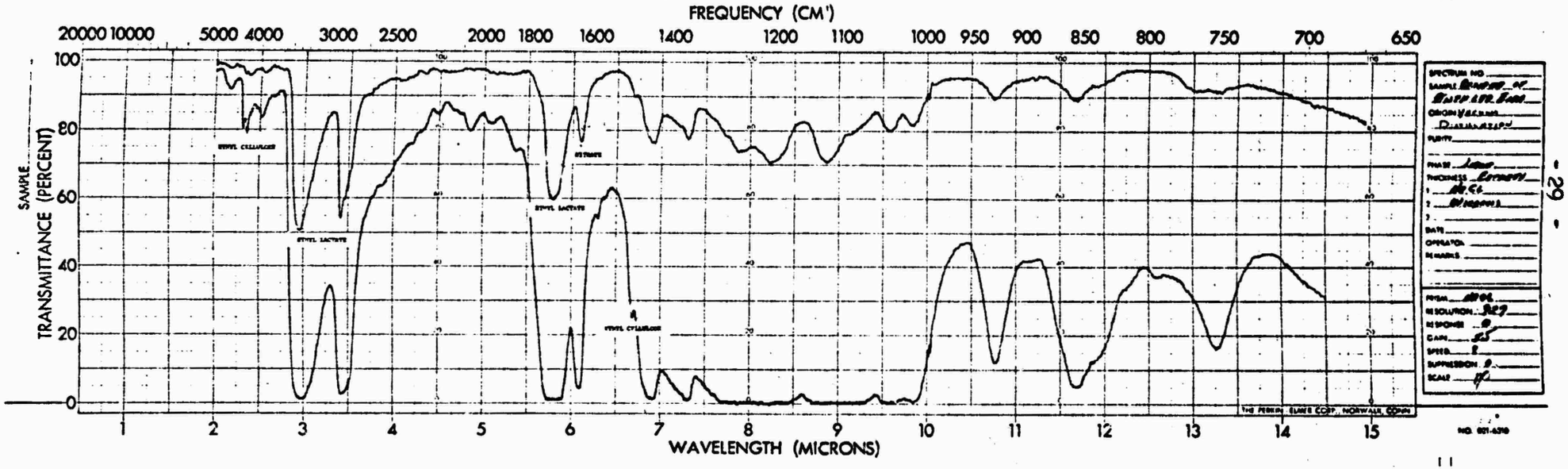
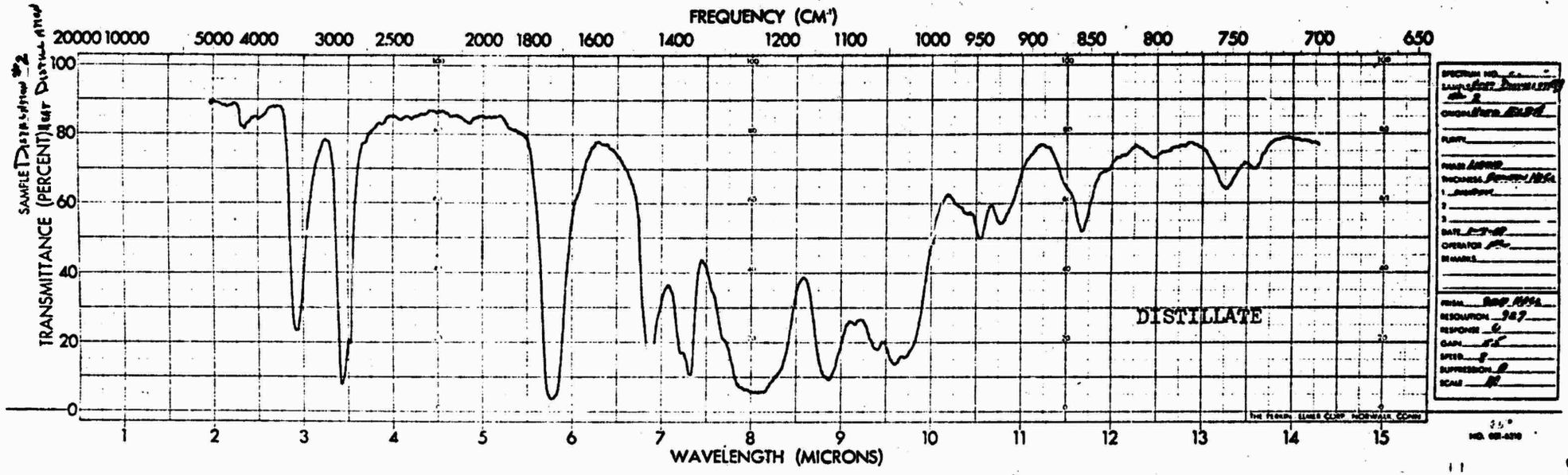


FIGURE 7

QUALITATIVE IR SPECTRA FOR FINAL VACUUM DISTILLATION RESIDUE



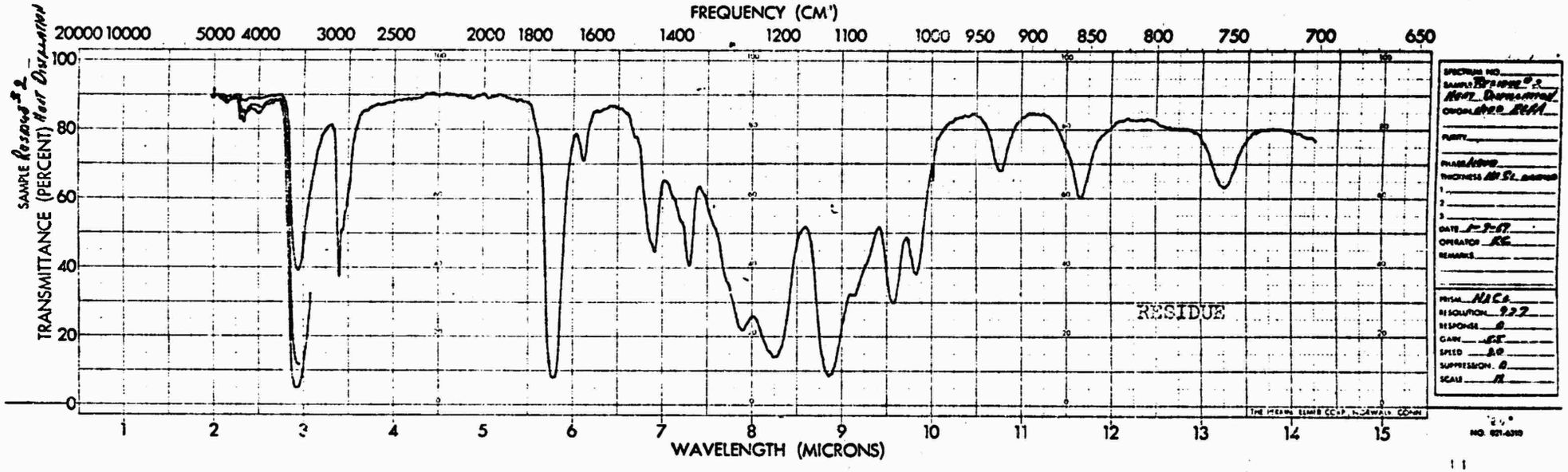


FIGURE 8

QUALITATIVE IR SPECTRA FOR AMBIENT DISTILLATION DISTILLATE AND RESIDUE

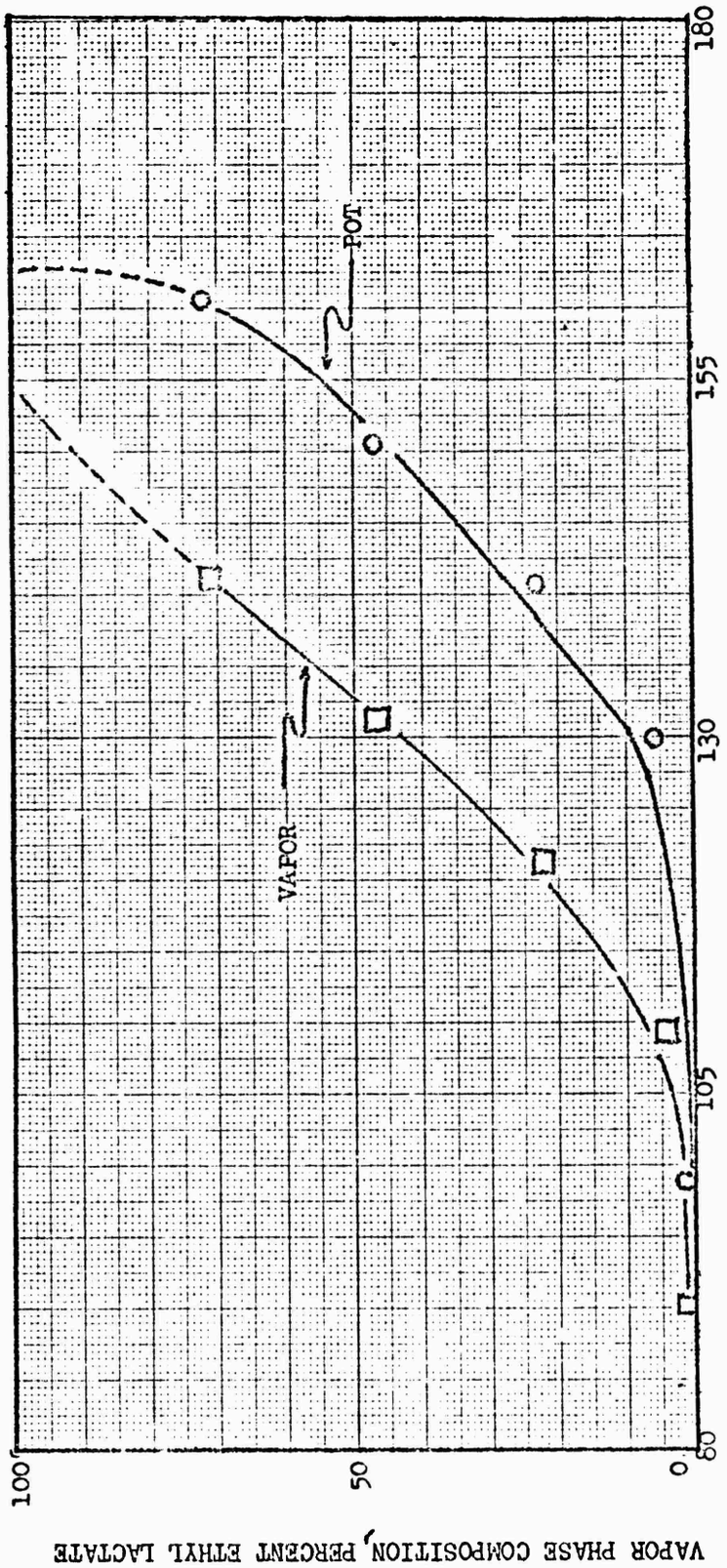


FIGURE 9

VAPOR PHASE COMPOSITION AS A FUNCTION OF TEMPERATURE

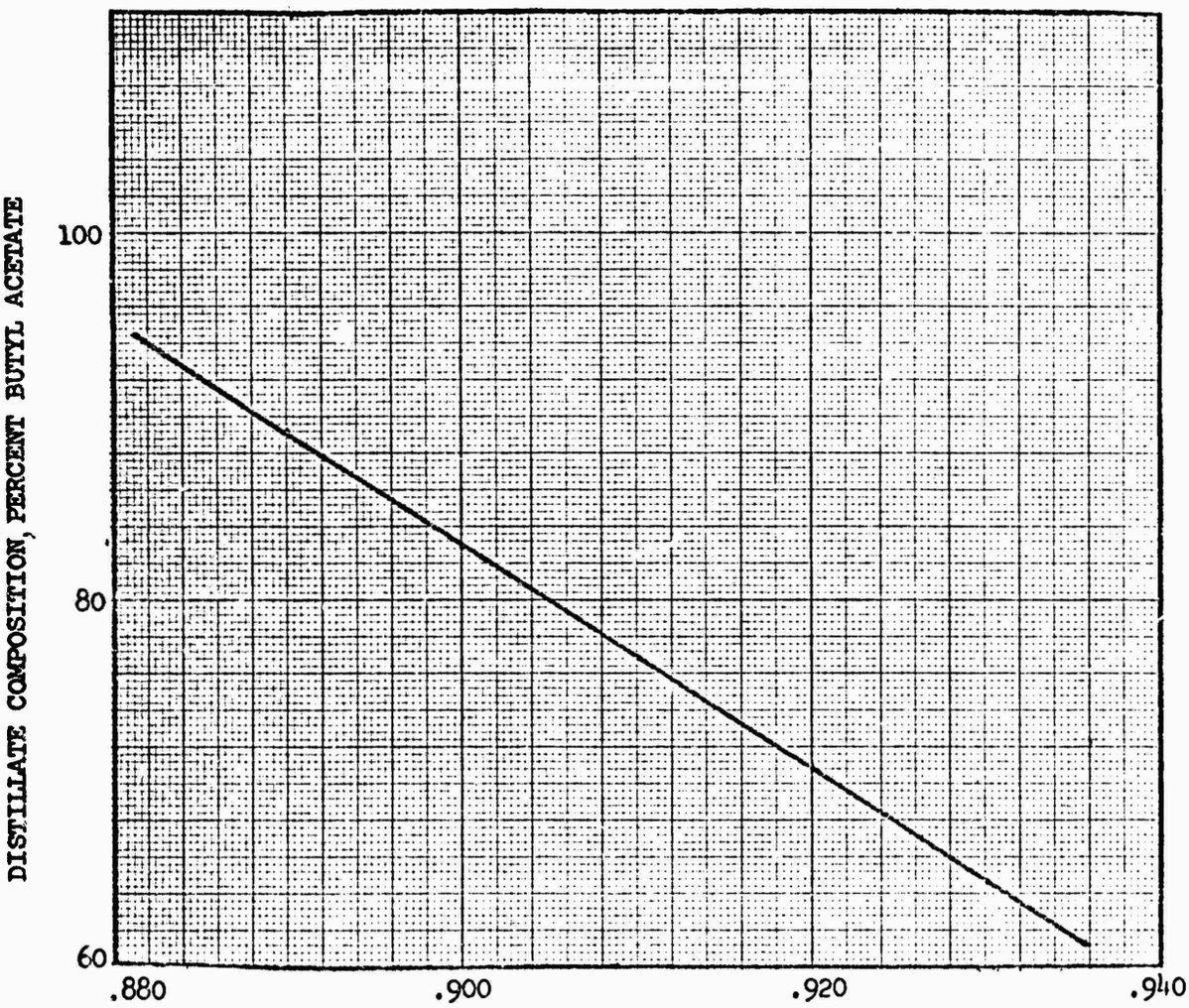


FIGURE 10

COMPOSITION OF DISTILLATE AS DETERMINED BY SPECIFIC GRAVITY

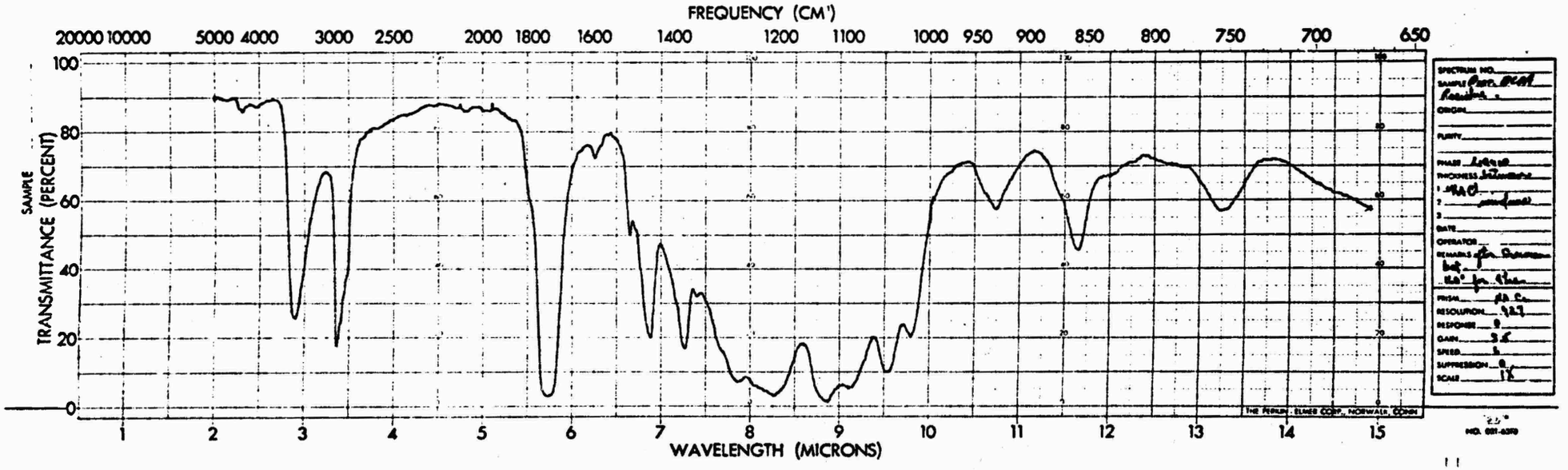


FIGURE 11

QUALITATIVE IR SPECTRA FOR DISTILLATION RESIDUE AFTER 4 HOURS AT 161°C

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13. ABSTRACT

Used ELBA solvent (Ethyl Lactate/Butyl Acetate) contaminated with components of N-5 propellant and water, was reclaimed by vacuum and ambient pressure distillation. Nitroglycerin (NG), water, and butyl acetate contents of the distillates are reported. Water separation in the distillate is discussed. Nitroglycerin content and thermal stability of the residues are reported. (U)

Distillations were performed successfully. Ninety percent recovery was obtained. Stability studies showed that the contaminated solvent could be distilled safely. (U)

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14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Inhibiting solvent for propellant						
Nitroglycerin - solvent distillation						
Double-base solid propellant.						